TECHNICAL NOTE

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Elemental and Microscopic Analysis in the 1993 Soft Drink/Syringe Product Tampering Incidents

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ABSTRACT: Throughout the summer of 1993, a large number of alleged product tamperings were reported to the US Food and Drug Administration concerning medical syringes and numerous other items found in carbonated soft drinks. In response to several of these claims, FDA's National Forensic Chemistry Center (NFCC) utilized microscopic and elemental analysis to help establish the veracity of the allegations. The techniques used include inductively coupled plasma atomic emission spectrometry, inductively coupled plasma mass spectrometry, stereoscopic light microscopy, scanning electron microscopy, and energy dispersive X-ray analysis. This report describes, in part, studies which were performed in response to some specific product tampering scenarios.

KEYWORDS: forensic science, elemental analysis, product tampering, inductively coupled plasma mass spectrometry, inductively coupled plasma atomic emission spectrometry, stereoscopic light microscopy, scanning electron microscopy, energy dispersive X-ray analysis

In the past 10 years, the number of reported product tamperings has increased substantially [*1–3*]. The Federal Anti-Tampering Act of 1983 [*4a*] includes sections which make false reports of a tampering and threatening a tampering, as well as actual product tampering, a crime. Threats and false reports of product tampering are punishable by up to five years in prison and a fine of $25,000. A conviction for product tampering can result in a prison sentence of three years to life with fines as high as $100,000 depending on whether injury or death occurred as a result of the tampering. Fines may be substantially increased as a result of the Fines Enhancement Statutes of 1985 [*4b*].

During the summer of 1993, the NFCC received items of evidence related to approximately 235 claims of tampering from around the country. A variety of items were allegedly found in cans of carbonated soft drinks including medical syringes, nails, pins, needles, bullets, and glass. Medical syringes were by far the most common item reportedly found. In many of these cases, the person reporting the incident eventually confessed to making a false report.

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There are three distinct times during which a tampering can occur: during production, during distribution and sale by breaching the container, or after the product is opened by the consumer. Thorough examination of the can surface, seam, closure, and lid can eliminate a breach of the container as the tampering mode. It was of prime importance in all cases to rule out, if possible, the bottling (canning) plant as a potential point of tampering. If it could be determined approximately how long a particular syringe was submersed in a can of soda, this information might provide evidence as to when an alleged tampering occurred. Coupled with information from the product code on the suspect can (which indicates the bottling date), the bottling process could be suspect or ruled out as a potential point of tampering. As part of NFCC's forensic investigation into some specific cases, studies were begun to evaluate 1) the potential for trace element contamination from the syringe found in the soda to provide an estimation of submersion time and 2) the analysis of the corrosive effect of soda on an aluminum crimp associated with one syringe type to provide an estimation of submersion time.

Trace element determinations have proven to be valuable forensic tools in several product tampering cases [*2,3*]. The ability to characterize forensic samples via trace elements has been used to help trace the source of a particular poison as in the case of cyanide-laced Tylenol capsules [*2*] and to discriminate glass fragments found in jars of baby food [*3*]. Inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) are powerful techniques for elemental analysis and have been used to characterize many forensic samples including glass fragments [*5*].

One potential source of trace element contamination in the soft drink samples was the rubber stopper from the syringe plunger. Zinc containing compounds have been used as vulcanization accelerators in rubber. Rubber stoppers from blood collection tubes have been found to be a source of Zn contamination in the analysis of blood [*6,7*] and rubber septa have been shown to cause Zn contamination in injection vials [*8*]. In the first part of this paper, a study using ICP-MS and ICP-AES describes the reproducible leaching of zinc over time from rubber stoppers by various soft drinks.

Stereoscopic light microscopy (SLM), scanning electron microscopy (SEM), and energy dispersive X-ray analysis (EDXA) of the corrosion points on a specific type of syringe after submersion in Diet Pepsi® is described in the second part of this study. One of the alleged tamperings involved a syringe which had an aluminum

crimp securing the stainless steel needle to the plastic syringe hub. Most of the syringe needles received were secured in plastic hubs with a plastic or epoxy material. A study was begun to determine the corrosive effect of Diet Pepsi® on this type of syringe needle. The corroded regions of the crimp were examined by stereoscopic light microscopy, scanning electron microscopy and energy dispersive X-ray analysis after specified submersion times in order to estimate the amount of time the suspect syringe had been submersed. Additionally, the Diet Pepsi® was analyzed for aluminum contamination by ICP-AES.

Experimental

Instrumentation

The inductively coupled plasma mass spectrometer used was a Fisons VG PlasmaQuad model PQ2+ Turbo with a Gilson model 222 autosampler. The operating conditions were as follows: forward power, 1350 W; reflected power, 0 W; coolant Ar flow rate, 14 L/min; auxiliary Ar flow rate, 1.1 L/min; nebulizer type, concentric TR-30-A3; nebulizer Ar flow rate, 0.82 L/min; sample uptake rate, 1 mL/min; water cooled spray chamber temperature, 5°C; analyzer stage vacuum, 1.6×10^{-6} torr; intermediate stage vacuum, $<10^{-4}$ torr; expansion stage vacuum, 1.5 torr; resolution, 0.8 amu; ion lens settings, typical. Data were collected in the peak jumping mode of the ICP-MS instrument with a dwell time of 10240 μs, 3 points per peak, and acquisition time of 1 min. The masses monitored were 54, 64, 66, 67, 68, 115, 118, 120, and 138.

The ICP-AES instrument used in this study was a Thermo Jarrell Ash model 61E Polyscan sequential/simultaneous unit. The operating conditions were as follows: forward power, 1150 W; coolant Ar flow rate, 14 L/min; auxiliary Ar flow rate, 1.0 L/min; nebulizer type, fixed cross flow: nebulizer Ar pressure, 30 psi; sample uptake rate, 1.5 mL/min. Data collection consisted of four 5-s integrations per sample. Zinc was determined in the simultaneous mode using the 213.9 nm line. Aluminum was determined in the sequential mode using the 308.2 nm line. Background correction was used in both the Zn and Al analyses.

The stereoscopic light microscope used was a Nikon SMZ-U stereoscope (~7 − 100X). Samples were illuminated by quartz halogen fiber optics.

A Leica S-260 scanning electron microscope equipped with a K&E backscattered electron detector and an Oxford Instruments eXL energy dispersive X-ray detector and analyzer were used. The SEM operating conditions were as follows: specimen chamber pressure, 1×10^{-5} torr; accelerating voltage, 15–18 kV; working distance, 22–30 mm; Analysis modes, secondary electron imaging (SEI), and backscattered electron imaging (BEI). For EDXA analysis, the following operating conditions were used: range, 0–20 keV; count rate, 3400–3600 cps; dead time, 22–50%; detector window, UTW (Ultra Thin Window); acquire time, 100 live seconds.

Zinc Leach Study: Methodology

A preliminary study was performed by submersing syringes in seven different soft drink products; Diet Pepsi®, Pepsi®, Diet Coke®, Coca Cola Classic®, Diet Sprite®, Sprite®, and Mountain Dew®. Aliquots from each of the seven products were analyzed by ICP-MS after 0, 1, 2, 3, 4–5, 7, 14, 21, and 28 days of submersion in a sealed container and by ICP-AES after 28 days.

A more controlled study was performed with 10 cans (12 oz.) of Diet Pepsi® obtained from several different lots. From each of the cans, 225 g of soda was placed into a 250-mL Nalgene bottle and the remainder into a 125-mL Nalgene bottle for use as a control. A Becton-Dickinson 1 cc insulin syringe was placed into each of the 250-mL Nalgene bottles. Prior to submersing the syringe in the soft drink, the needle guard was discarded; and the plunger was separated from the syringe body. The needle and stopper were pointed to the bottom of the bottle. Five mL aliquots from each of the samples and controls were analyzed by ICP-MS after 0, 1, 3, 8, 14, 22, 28, 43, and 150 days. ICP-MS results were corrected for instrument drift using an In internal standard at a concentration of 10 ng/mL. Zinc concentrations were corrected for volume changes as aliquots were removed from the sample solutions. In addition, the data presented has been normalized to reflect the volume of a full can of soda (354 mL). In addition, one rubber stopper each from a Becton Dickinson 1 cc Allergy Syringe was submersed into one Diet Coke® and one Diet Pepsi® sample. Aliquots from each of these studies were analyzed by ICP-MS after 0, 4, 7, 14, 21, and 152 days.

In a separate study, 3 different kinds of 1 cc syringes were submersed in Diet Coke® and/or Diet Pepsi®. In each of these cases, the plunger and rubber stopper were left either partially or completely inside the syringe body. The syringes used were the same types encountered in some of the alleged tampering cases which included Becton-Dickinson 1 cc allergy syringes, Monoject® 1 cc insulin syringes and Terumo® 1 cc insulin syringes. Aliquots from each of these studies were analyzed by ICP-MS after 0, 4, 7, 14, 21, and 152 days.

Zinc Leach Study: Sample Preparation and Analysis

Aliquots of diet soft drink were analyzed directly with no preparation other than the addition of a 10 ng/mL In internal standard. However, as the time studies progressed, it was necessary to dilute samples by a factor of 5 with DDW in order to keep the Zn concentration within the linear range of the pulse-counting mode of the ICP-MS.

Regular (sugar-containing) soft drinks were diluted by a factor of two with DDW in order to minimize matrix effects from the sugar in the ICP-MS analysis. A 10 ng/mL In internal standard was used. The ICP-MS instrument was calibrated daily using a 1% nitric acid blank and a 10 or 100 ng/mL standard of Fe, Zn, Ba, and Sn in 1% nitric acid. Standard and blank check samples were analyzed after every 10 samples. The ^{66}Zn isotope was used for ICP-MS quantitation.

Al Crimp Study: Methodology

In this study, two complete syringes with 22 gauge aluminum crimp needles (Monoject®) were placed in each of eight 20-fluid ounce bottles of Diet Pepsi® with plastic screw-type caps. One syringe had the plunger inside the barrel and the other had the plunger removed from the barrel. Both syringes were inserted with the needle pointed down. Each bottle was sealed and gently agitated to permit the barrel of both syringes to fill with soft drink product. Two syringe needles with aluminum crimps from the same lot of syringe needles were selected as control needles and were not submersed in Diet Pepsi®. The syringes were removed from the bottles after 1, 2, 3, 4, 9, 10, 14, and 21 days. For the purposes of this study, the needle crimps from 1, 14, and 21 days along with control (no submersion) crimps were analyzed by SLM, SEM, and EDXA. The Diet Pepsi® was analyzed for aluminum by ICP-AES after the syringe needles were removed. Aliquots of Diet

Pepsi® were analyzed from bottles with syringe submersion times of 1, 2, 3, 4, 9, 10, 14, and 21 days.

Aluminum Crimp Study: Sample Preparation and Analysis

After the syringes were removed from the Diet Pepsi® bottles, any damage in the form of pitting and/or loss of crimp material was photodocumented. The syringes were air dried for approximately 24 hours, and stereoscopic light microscopic analysis was performed. The syringes were not washed prior to initial analysis. A blunt stainless steel probe was then used to gently break the corrosion "flowers" from the corroded areas of the crimp. The partially cleaned needle assembly was placed (hub, crimp and needle) into a 30 mL glass scintillation vial filled with filtered, distilled deionized water. The needle assembly was soaked for two minutes and then placed in a gentle ultrasonic bath for two minutes with occasional swirling to dislodge remaining pieces of dried soft drink and corrosion residue. The needle assembly was then dried with a high-velocity, filtered air.

The cleaned and dried needles were analyzed in the SEM. The SEM analysis was limited to the crimp region. The needle/hub assembly was secured in a micro vise inside the specimen chamber of the SEM to permit analysis of the crimp region. Each needle was found to have four crimp indentations oriented at 90° angles to each other. Both secondary electron images (SEI) and backscattered electron images (BEI) were prepared for each of the four positions (indentations). Following SEI and BEI image preparation, EDXA analysis was performed on each crimp position. Seven elements were detected using spot mode X-ray analysis. Oxygen, carbon, aluminum, silicon, sulfur, phosphorus, and copper X-ray mapping was performed to show the locations of previously identified elements on the surface of the crimp regions. The X-ray mapping was specific for each of the seven elements.

Aliquots of Diet Pepsi® were analyzed directly for aluminum by ICP-AES. A 2-point calibration was established for Al using a 2% nitric acid blank and a 1 μg/mL Al standard.

Results and Discussion

Zinc Leach Study Results

The first situation described in this report involves an insulin syringe allegedly found in a can of Diet Pepsi®. The plunger and rubber stopper were separated from the syringe barrel. ICP-MS and ICP-AES were used to measure the concentration of Zn leached from rubber stoppers submersed in Diet Pepsi® and other soft drinks for periods as long as 5 months. Preliminary experiments indicated that Fe, Sn, and Ba might also be useful markers. As a result the concentration of these elements was also monitored.

In the preliminary work, five of seven different soft drink types showed the same general trend; zinc concentration increased with time over the 28 day study. Figure 1 demonstrates how Zn concentration was affected for the 7 sodas over the 28 day period. It is interesting that except for Pepsi® and Coca Cola Classic®, there is a significant increase in Zn concentration after 7 to 14 days of submersion. The experimental parameters in this initial study were not stringently controlled; therefore, it is difficult to attach significance to the difference in Zn concentrations between the 21st and 28th days of the study. It is not understood at this time why the two regular colas, Pepsi® and Coca Cola Classic® did not show an increase in Zn concentration over the 28 day period. No significant changes in Sn, Fe, or Ba concentrations were noted during the study.

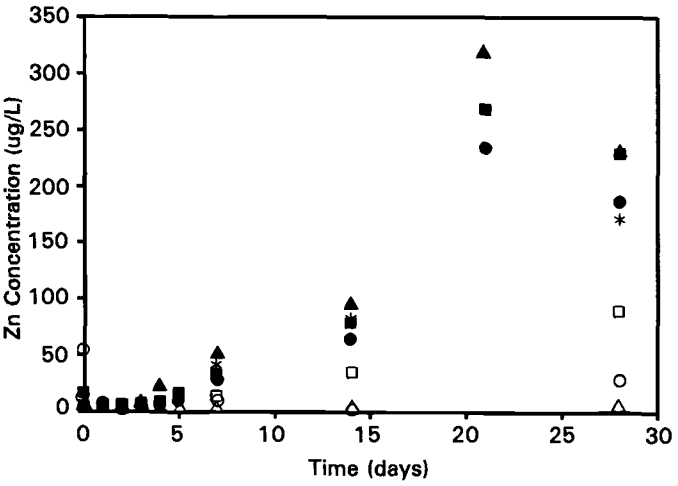

FIG. 1—*Zn concentration in carbonated soft drinks versus syringe submersion time.* ▲ Diet Pepsi, ● Diet Coke, ■ Diet Sprite, * Mountain Dew, □ Sprite, △ Pepsi, and ○ Coca Cola Classic.

A confirmatory analysis for Zn by ICP-AES was completed on Diet Pepsi, Diet Coke, and Diet Sprite with controls and samples after 28 days of exposure. Table 1 shows that the results of this analysis compare well with the ICP-MS data. Other elements detected by ICP-AES include Ba, Ca, P, Mg, Na, Si, Sr, and K; however, there was no significant difference in concentration between controls and 28 day exposure samples.

In the second part of the study, each of 10 Diet Pepsi samples showed an increase in Zn concentration with syringe submersion time. Table 2 lists the average, standard deviation and 95% confidence limit for the 10 samples over a period of 150 days. It is interesting to note that after 3 days of exposure, the Zn concentration approximately doubled; and after 8 days the Zn concentration

TABLE 1—*Comparison of ICP-AES and ICP-MS results.*

Sample Identification, and Exposure Time		ICP-AES Zn (μg/L)	ICP-MS Zn (μg/L)
Diet Pepsi Control	0 days	8	3.6
Diet Pepsi	28 days	246	231
Diet Coke Control	0 days	9	3.8
Diet Coke	28 days	193	187
Diet Sprite Control	0 days	17	14
Diet Sprite	28 days	211	231

TABLE 2—*ICP-MS results for Zn leaching experiment with Diet Pepsi.*

Time (days)	Average Zn (n = 10) (μg/L)	Standard Deviation (μg/L)	95% Confidence Limit (μg/L)
0	4.1	2.0	0.7
1	3.6	1.8	0.7
3	9.2	2.9	1.1
8	44	5.0	1.9
14	90	9.6	3.6
22	148	15	5.6
28	187	20	7.5
43	343	31	12
150	747	40	15

has increased by approximately an order of magnitude. Figure 2 graphically shows the increase in Zn concentration with time for these samples.

The source of the zinc contamination was confirmed to be the rubber stoppers by submersing a rubber stopper from a 1 cc syringe in each of two diet colas. In these samples, Zn concentration increased with time in essentially the same fashion as described above. A significant increase in Zn concentration was noted after 7 days of exposure.

In an additional study, Zn concentration remained essentially unchanged over a period of 152 days in each case where the plunger was located fully or partially inside the body of the syringe. No increase in Zn concentration was observed. It is important to note that Zn was only leached when the plunger was separated from the syringe body and the stopper was fully exposed to the soft drink.

If sufficient time has elapsed between bottling and the alleged discovery of a medical syringe with separated plunger in a canned diet soft drink, zinc concentration should be elevated. Note however that an elevated zinc concentration does not "prove" that the syringe was submersed in the soft drink for an extended period of time unless other sources of zinc contamination can be eliminated. Depending on bottling and discovery times, if no difference in zinc concentration is found between the allegedly tampered product and a control sample with the same product code, it can be concluded that the tampering did not occur during the bottling operation.

Aluminum Crimp Study Results

The second situation described in this report involves the corrosive effect of Diet Pepsi® on the type of aluminum crimp found in an alleged tampering case. SLM, SEM, and EDXA were used to determine the location and extent of corrosion in syringes removed at 24 hour, 14 day, and 21 day intervals. It should be noted that aluminum soft drink cans have a protective polymer coating on the inside to prevent any interaction between the aluminum can and the product. Visual and stereoscopic light microscopy analysis of the crimp regions of the study needles showed light brown and raised sites of corrosion with the amount of corrosive material related to the length of submersion time. Usually there was only one site of corrosion on each aluminum crimp.

Four positions (sides) of each crimp were analyzed using SEM and EDXA. The control aluminum crimp (Fig. 3) and study aluminum crimps from 24 hour, 14 day, and 21 day submersion in Diet Pepsi® (Figs. 4, 5, and 6, respectively) all show one of four crimp indentions. The three submersion crimps all show the position with a single region of corrosion. SEI analysis of each crimp position revealed surface irregularities including processing striations and shallow pitting on all needle crimps. The submersion crimps showed additional features including raised or blistered

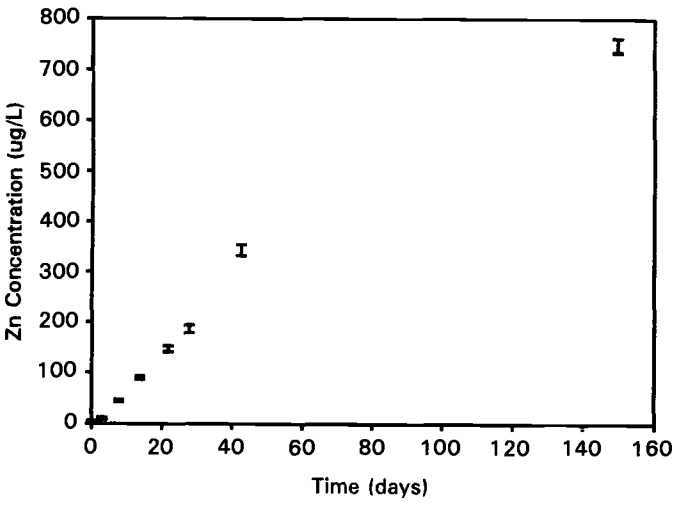

FIG. 2—*Zn concentration in Diet Pepsi versus syringe submersion time as determined by ICP-MS.*

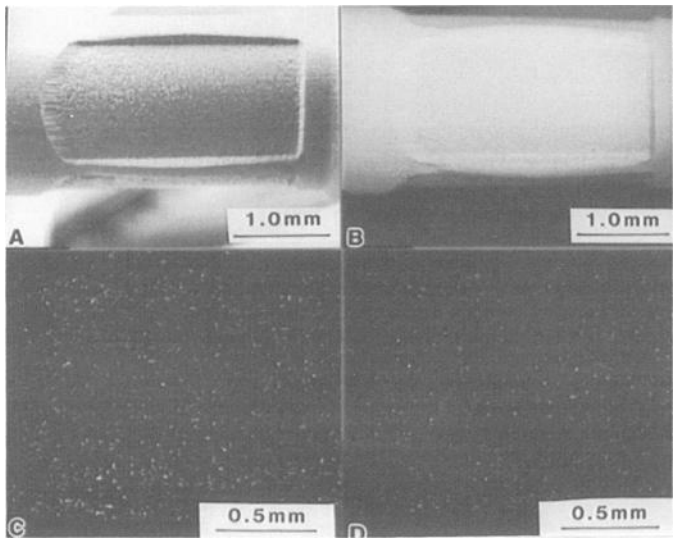

FIG. 3—*Control aluminum crimp from a syringe needle not exposed to Diet Pepsi®: secondary electron image* (A); *backscattered electron image* (B) *showing no evidence of surface damage; oxygen X-ray map* (C), *and phosphorus X-ray map* (D) *showing no X-ray production for either element.*

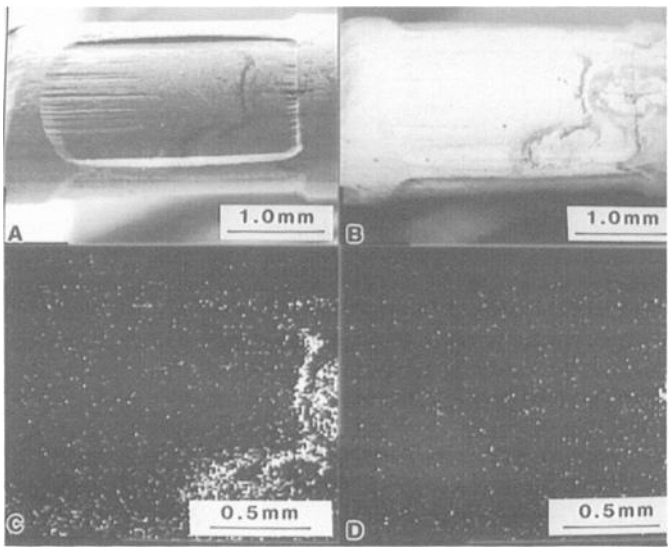

FIG. 4—*Aluminum crimp from a syringe needle exposed to 24 hour soaking in Diet Pepsi® with a single site of corrosion: secondary electron image* (A); *backscattered electron image* (B); *oxygen X-ray map* (C), *and phosphorus X-ray map* (D) *showing an increase in X-ray production at the site of corrosion.*

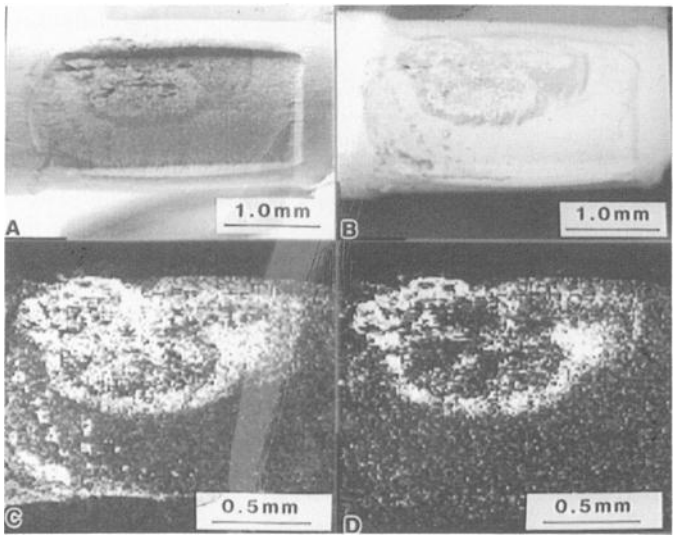

FIG. 5—*Aluminum crimp from a syringe needle exposed to 14 days soaking in Diet Pepsi® showing increased corrosion at a single site: secondary electron image* (A); *backscattered electron image* (B); *oxygen X-ray map of crimp* (C), *and phosphorus X-ray map* (D) *showing a notable increase in X-ray production for both elements.*

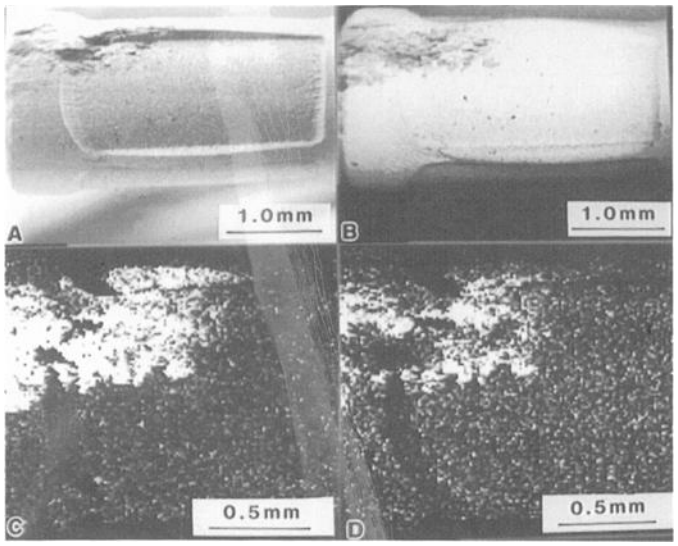

FIG. 6—*Aluminum crimp from a syringe needle exposed to 21 days soaking in Diet Pepsi® showing still increasing corrosion, deep pitting as well as loss of metal at a single region on the crimp, secondary electron image* (A); *backscattered electron image* (B); *oxygen X-ray map* (C), *and phosphorus X-ray map* (D) *of crimp showing high elemental X-ray production for both elements at the corrosion site.*

surface regions (Fig. 4-A) to deeper pitting and eventually severe corrosion and loss of metal on the aluminum surface (Fig. 6-A) that were directly dependent on the length of submersion time. BEI images showed the loss of BEI signal on the crimp regions directly coincided with the regions of surface corrosion. The BEI is directly dependent upon the atomic density of the analyzed sample (that is, the higher the atomic density, the brighter the image). Since the BEI production from the corrosion regions was darker than the surrounding aluminum crimp, the dark region would have an atomic density less than aluminum and consequently composed of elements having an atomic number less than that of aluminum.

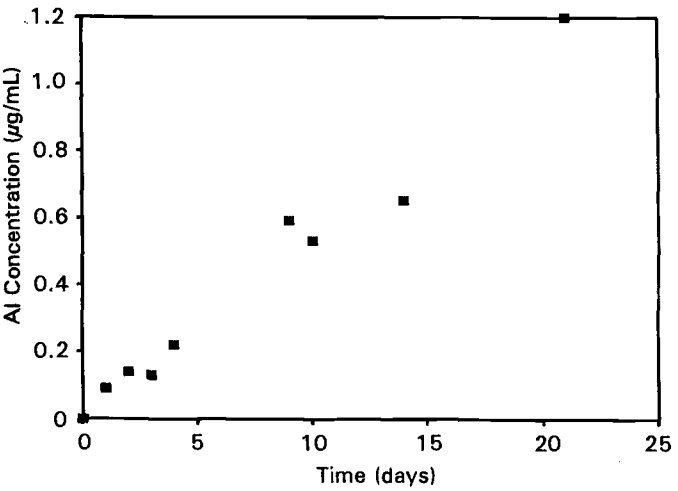

FIG. 7—*Aluminum concentration in Diet Pepsi® versus syringe submersion time as determined by ICP-AES.*

SEM/EDXA analysis in the form of X-ray mapping revealed that oxygen was identified at each corrosion site. The density of the oxygen X-ray production was directly related to and increased with longer submersion time in Diet Pepsi®. Oxygen X-rays were found to directly coincide with the darker regions on the BEI. The loss of aluminum X-ray production at the corrosion site shows the corrosion region was being "blocked" by another element(s). The high oxygen X-ray density at the corrosion sites would suggest the presence of oxides. In addition, both the phosphorus and (to a lesser degree) sulfur X-ray production was also directly related to increased submersion time in Diet Pepsi® and corresponded with the regions of deep corrosion. Figures 3, 4, 5, and 6 all display the oxygen X-ray map at the lower left or "C" quadrant and the phosphorus X-ray map at the lower right or "D" quadrant. The bright regions on the X-ray maps correspond directly with the X-ray production of the selected element on the surface of the aluminum crimp and the region of corrosion. The X-ray maps were produced at approximately two times the magnification of the SEI/BEI images at the site of observed corrosion. The absence of carbon X-ray density points (not shown) suggest all soft drink sweetener and caramel coloring residue had been removed during the cleaning process prior to SEM/EDXA analysis. It further indicates that the detected oxygen was probably bound to the crimp metal, again suggesting aluminum oxide.

Aliquots of the Diet Pepsi® samples used in the crimp study were analyzed for aluminum by ICP-AES. The limit of detection was determined to be 0.06 µg/mL Al. Aluminum was not detected in the control Diet Pepsi®. After 1 day, the Al concentration was approximately 0.1 µg/mL. The Al concentration increased to approximately 1.2 µg/mL after 21 days. A plot of Al concentration versus length of submersion time is shown in Figure 7. Because Al concentration increased fairly linearly with submersion time, analysis of any remaining Diet Pepsi® from an alleged tampering could provide a valuable investigative lead.

Conclusions

Microscopic and elemental analysis were used extensively during the 1993 soft drink/medical syringe tamperings. Zinc leached from the rubber stopper of a medical syringe by a carbonated soft drink such as Diet Pepsi® can be used as an indication of how long that particular syringe was submersed in the soft drink provided that

the rubber stopper has been separated from the syringe barrel and allowed to soak in the soft drink. This information provides evidence regarding when an alleged tampering could have occurred. However, it was disappointing that in cases where the plunger and stopper were left inside the body of the syringe, no change in Zn concentration with time was noted. In a similar study, microscopic analysis of the corroded region of an aluminum crimp from a medical syringe also provided evidence related to when an alleged tampering could have occurred.

Both studies can be used to eliminate the bottling plant as a potential place where the alleged tamperings could have occurred. In addition, information regarding how long a particular syringe was submersed can be used to provide investigative leads as to when and where a tampering could have occurred. It should, however, again be noted that neither the presence of zinc in the soft drink nor corrosion on the aluminum crimp of a medical syringe "proves" that a tampering has occurred.

Acknowledgment

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